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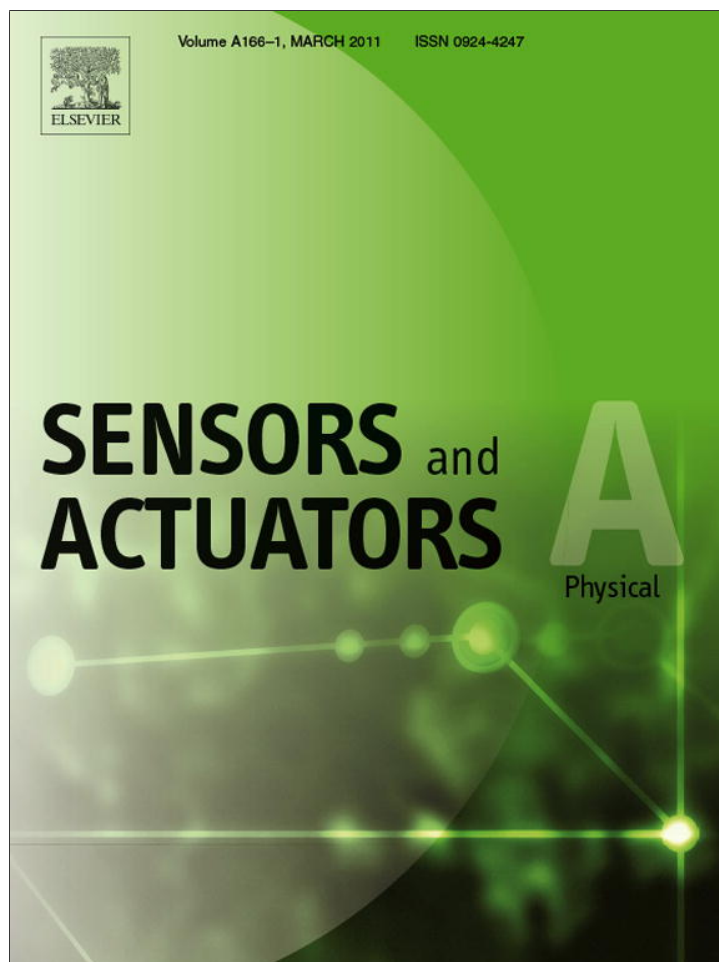
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Optimization of nickel nanocomposite for large strain sensing applications

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ABSTRACT

A novel large strain sensor has been developed using a silicone/nickel nanostrand/nickel coated carbon fiber nanocomposite system. The effect of conductive filler volume fraction on the piezoresistive response of the nanocomposite sensor has been studied in order to determine the optimal composition for use in large strain/motion sensing applications. Electromechanical testing of various compositions revealed that optimum performance was achieved using 11 vol% nickel nanostrands with 2 vol% nickel coated carbon fiber in the silicone matrix. Initial results indicate that this nanocomposite is capable of sensing strains of over 40% elongation.

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1. Introduction

The sensing of mechanical strain is integral to many engineering applications such as ballistic testing, biomechanics (e.g. advanced prosthetics), haptic interfaces, structural health monitoring, etc. However, at present, most conventional strain gauges can only measure strain reliably up to a few percent [1]. Several methods, such as interferometric strain/displacement gauges (ISDG) [2], digital image correlation (DIC) [1], and differential digital image tracking (DDIT) [3] have been developed in order to be able to measure larger strains; however, such methods are expensive, complicated and cumbersome. There is an obvious need for a simple, inexpensive strain gauge technology that could be easily integrated into the many engineering designs in which large strains must be sensed.

Conductive polymer composites (CPCs) have shown promise as inexpensive large strain sensors [4–7]; however, their use has been limited due to their prohibitive lack of reproducibility [8,9]. A candidate large strain sensor will have the following characteristics: (1) an obvious ability to sense strains in excess of those capable using conventional methods; (2) high measurement fidelity; and (3) good repeatability between sensors. The objective of this study was to design a sensor capable of measuring strains of $\epsilon_{\max} \geq 40\%$ elongation. In order to ensure high measurement fidelity and

repeatability, focus was placed on minimization of the measurement uncertainty due to measurement error between sensors (ϵ_{err}). Silicone/nickel nanostrand (Si/NiNs) composites have been shown to be effective at measuring strains in excess of 40% elongation [6]. Their exponential piezoresistive response provides high measurement resolution. At the same time their elastomeric matrix and low Young's modulus (~ 6.5 MPa) allow them to deform to large strains without significantly stiffening the substrate to which they are attached. However, due to the percolative nature of charge transport in CPCs, Si/NiNs nanocomposites have an idiosyncratic critical strain, ϵ_c , below which their piezoresistive response is nearly zero. In order to improve the material response of this nanocomposite system such that it becomes viable for use as a large strain sensor, a study was conducted to examine the effect of filler volume percentage on the piezoresistive response of the material. As part of this study the effect of the addition of a second conductive filler particle of a distinct length scale, namely nickel coated carbon fibers (NCCFs), was investigated. The optimized composition was shown to be capable of sensing strain in excess of 40% elongation while showing significant repeatability between gauges. This initial optimization of a novel Si/NiNs/NCCF nanocomposite material demonstrates the great potential of this material for use as an inexpensive strain gauge technology that can measure strains greater than 8 times those of conventional gauges.

2. Theory

In previous work, tensile testing of various elastomeric materials filled with NiNs demonstrated the extreme piezoresistive

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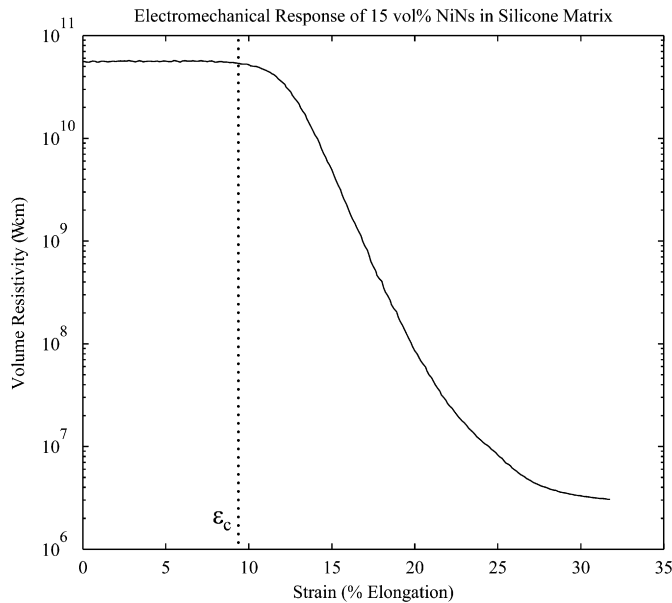


Fig. 1. Typical piezoresistive response of Si/NiNs composite.

response of elastomer/NiNs composites [6]. However, the potential applicability of these previously tested composite systems to strain sensing applications was limited by their percolation like response. As these composites were pulled in tension, an effective critical strain, ϵ_c , was observed. Once ϵ_c was exceeded the system would begin to respond, similar to the critical volume fraction in percolation type systems (Fig. 1) [10]. In the elastomer/NiNs composites previously studied, ϵ_c typically occurred between 10% and 20% elongation.

In traditional percolation theory, a system is represented by an undirected graph of nodes and connecting lines. The lines in this lattice are called 'bonds' and the nodes or intersections of the lines are called 'sites'. For a particular simulation, sites (in the case of site percolation) or bonds (in the case of bond percolation) are either closed or open with a given probability, p . As p increases, clusters of closed sites or bonds will develop and one can define a critical or threshold probability, p_c , which is equivalent to the number fraction of closed sites or bonds required to create a cluster that spans the lattice. This percolation threshold is intimately related to the structure of the graph. The graph structure of traditional percolation theory is that of a regular lattice. However, the intrinsic structure of some physical systems does not lend itself well to an ordered lattice representation. For cases of this type continuum percolation theory is used. In continuum percolation theory the locations of sites are not limited to discrete points on the lattice; rather, they are allowed to exist at any point in the continuous domain. This allows for the modeling of random networks, such as the one under consideration.

Consider for a moment the problem of electrical conductivity for a CPC in the context of a continuum bond percolation model. In this case the nodes represent the conductive phase and the bonds represent conductive paths through the non-conductive phase (due to, e.g. tunneling, electron hopping, etc.). When a bond is open there is no conduction between adjacent nodes, when it is closed there is conduction between nodes. In the case of Si/NiNs composites this is thought to be due to quantum mechanical tunneling, in which case a bond is open only if the conductive phase particles are within ~ 1 nm of each other [11]. We note that in this formulation each conductive phase particle is reduced to a point on the percolation graph, and the bonds connect conductive phase particles only along the shortest path between particles (i.e. not centroid to centroid). If a representative element or 'unit cell' of a given graph is examined (Fig. 2) the threshold probability for conduction can be deduced. For a conductive path to exist through a node at least two of the bonds connected to that node must be closed. It would then be expected that the threshold probability be approximately equal to the number of required closed bonds divided by the total number of available bond locations. For the honeycomb lattice in Fig. 2 one would expect a critical probability of $p_c \approx 2/3$, for the square lattice $p_c \approx 1/2$, and for the triangular lattice $p_c \approx 1/3$ and indeed these values match well with those reported in the literature [12]. Fig. 2 also shows a representative 'unit cell' for a continuum model where the central node is connected to 6 adjacent nodes, for which we would again expect $p_c \approx 1/3$. This is to say that by increasing the number of bonds attached to each node the percolation threshold can effectively be lowered. This fact can be exploited to manipulate the critical strain at which Si/NiNs nanocomposites begin to respond piezoresistively. By some means, as this material is strained the spatial distribution of NiNs with respect to one another (which directly corresponds to the distribution of bond lengths in the continuum percolation graph) is altered such that the material passes from a sub-critical percolation regime through the percolation threshold and into a highly conductive super-critical regime. By increasing the initial (unstrained) number of potential paths between conductive phase nanoparticles the threshold (ϵ_c) can be lowered and the amount of strain required to cross the threshold should be less. Increasing the proximity of conductive phase particles for a static system (i.e. one that is not subjected to mechanical loading) is typically accomplished by increasing the volume fraction of the conductive phase. However, in nanocomposites this strategy can require prohibitively large quantities of expensive nanoparticles. An alternative method to modify the initial bond length distribution is to introduce a second conductive phase of a distinct length scale—effectively superimposing the two respective percolation graphs. The effect of this is to create 'super-nodes' or 'super-sites' in the composite graph that connect small clusters and create larger clusters at lower volume fractions (Fig. 3).

The creation of these 'super-nodes' significantly reduces the threshold probability as can be seen from Fig. 4, where a 'super-node' connecting 50 clusters is schematically represented. The expected threshold for this case would be $p_c \approx 1/25$.

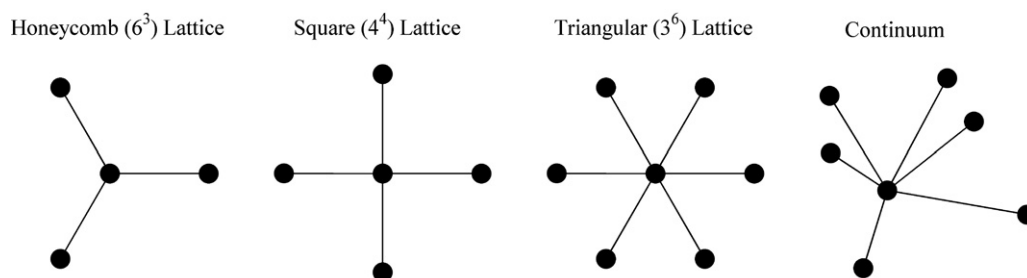


Fig. 2. Several typical lattices for percolation simulations.

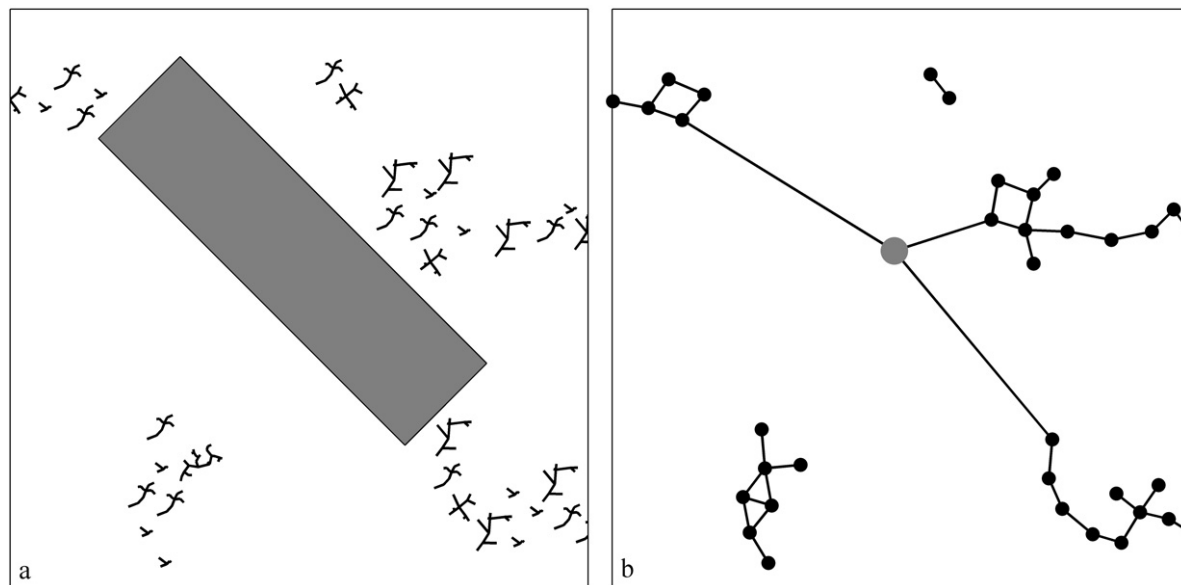


Fig. 3. Schematic of the creation of 'super-nodes' by the inclusion of a second phase of distinct length scale. (a) A 2D representation of the distribution of NiNs (thin black bifurcated structures) and NCCF (gray rectangular structure) in the silicone matrix. (b) Continuum percolation representation of (a), where black sites (black circles) represent discrete NiNs, gray sites (gray circles) represent NCCFs, and bonds (black lines) represent conductive junctions (i.e. locations where the distance between conductive phase particles is ~ 1 [11]).

While the introduction of this larger second phase does increase the volume fraction of conductive phase material (with fewer and less expensive particles), the most important effect is that of manipulating the bond length distribution such that the average distance between conductive phase domains is decreased. As the material is mechanically loaded *this* is what directly causes the consequent decrease in ε_c . There is, however, a competing increase in composite stiffness because of the lower compliance in the larger phase, which is a limiting factor for ε_{\max} . Therefore, this study is couched as an optimization problem in which simultaneous minimization of ε_c and maximization of ε_{\max} is sought. The above theory as well as prior work suggests that by finding the ideal volume fractions of the nano-phase and the larger macroscopic phase conductive fillers the spatial distribution of conductive phase particles can be indirectly optimized so as to achieve this objective. In order to

ensure the usefulness and applicability of the resulting nanocomposite material as a large displacement strain sensor, the metric of ε_{err} is also used to identify the ideal composition. In previous electromechanical testing of Si/NiNs composites there was little to no response manifested with NiNs volume fractions below 7%. Conversely, mechanical instability was observed at volume fractions above 15%. These previous results guided our selection of the following compositions for testing:

7% NiNs + 4% NCCF

9% NiNs + 1% NCCF

9% NiNs + 2% NCCF

9% NiNs + 3% NCCF

11% NiNs + 1% NCCF

11% NiNs + 2% NCCF

where filler content was measured in volume percentage. The following compositions were also tested: 7% NiNs + 1% NCCF, 7% NiNs + 2% NCCF, and 7% NiNs + 3% NCCF. However, there was limited piezoresistive response and therefore the data is not included in this study. Three trials were performed for each of the compositions that were tested; however, for both the 9% NiNs + 1% NCCF and 9% NiNs + 2% NCCF compositions the data for one of the trials was too noisy and differed significantly from the other two trials and was consequently thrown out.

3. Experimental procedures

3.1. The mold

Small 'dog-bone' tensile samples were prepared using an aluminum mold. To accommodate both small and large batches each slot in the mold was made for a set of three dog-bone samples

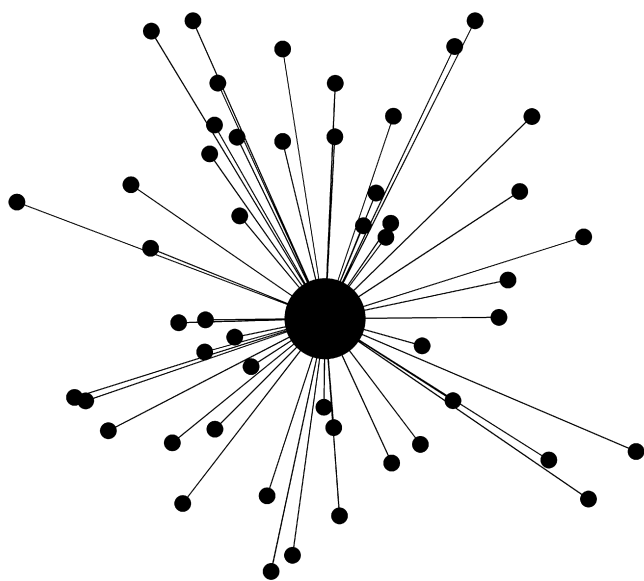


Fig. 4. Schematic 'unit-cell' for a 'super-node'.

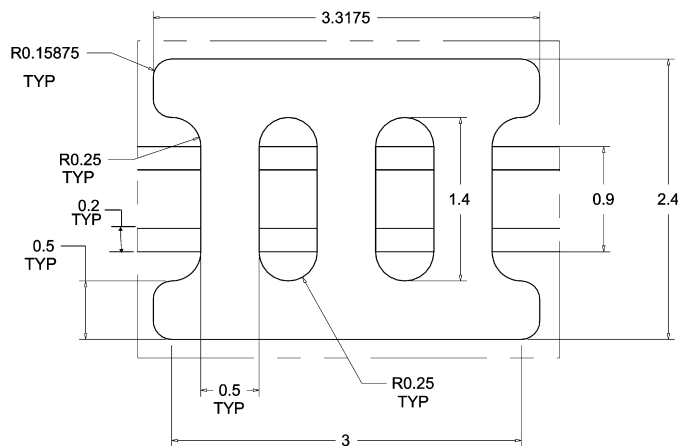


Fig. 5. Dog-bone mold schematic (units in cm).

(Fig. 5). Channels were cut into the mold along its length so that copper wires could be cast into the dog-bone samples for improved and consistent electrical contact (Figs. 5 and 6). The mold was treated with an aerosol polytetrafluoroethylene (PTFE) mold release in order to facilitate post-cure removal of the nanocomposite samples.

3.2. Nanocomposite preparation

The matrix material for the Si/NiNs/NCCF nanocomposite in this study was Dow Corning's® two part silicone elastomer, Sylgard 184. The principal conductive filler that was used was nickel nanostrands. NiNs are high aspect ratio nanoparticles with a unique bifurcated structure [6,7,13,14]. They are made by Conductive Composites, LLC through a proprietary low temperature, atmospheric pressure chemical vapor deposition (LTAPCVD) process [13]. For the larger second phase, carbon fibers were used, which were coated

with 20 wt% nickel (~80 nm thick) and cut to lengths of about 2 mm.

The silicone base was mixed with the NiNs in a planetary centrifugal mixer in order to achieve uniform dispersion of the nanoparticles. The Si/NiNs solution was then screened through a 40 gauge mesh to eliminate any large particles that may have infiltrated the solution and to provide some degree of uniform particle size. To this concentrated solution were added the NCCF, additional silicone base, and the cross-linking catalyst. With increasing filler volume percentage it became necessary to add small amounts of solvent as well, in order to decrease the viscosity sufficiently such that proper mixing could occur and to decrease shear stresses that could destroy the unique structure of the NiNs particles. This mixture was then mixed again in a planetary centrifugal mixer.

After mixing, the solution was then placed in the mold using a metal spatula. This method of filling the mold introduced air bubbles into the samples. Therefore, in order to prevent porosity in the cured samples, they were degassed under vacuum at room temperature. The samples were then cured at 100 °C overnight.

3.3. Testing

Mechanical testing was performed on the samples using an MTS 880 Material Test System. The metallic clamshell grips that were used were insulated from the rest of the test system using custom made Phenolic spacers. In order to prevent slippage, an abrasive covering was applied to the grips. Each sample was connected in series with a $120 \Omega \pm 0.01\%$ precision resistor. This voltage divider circuit was shielded and excited by a 10 V DC source. The samples were pulled in tension at a strain rate of 2.5 mm/min until fracture. Strain was calculated from the displacement of the test system cross-head and the gauge length of the samples.

As each sample was pulled, the voltage drop across it decreased. The resistivity of the sample was calculated from the change in voltage using the following formula, derived from the voltage divider

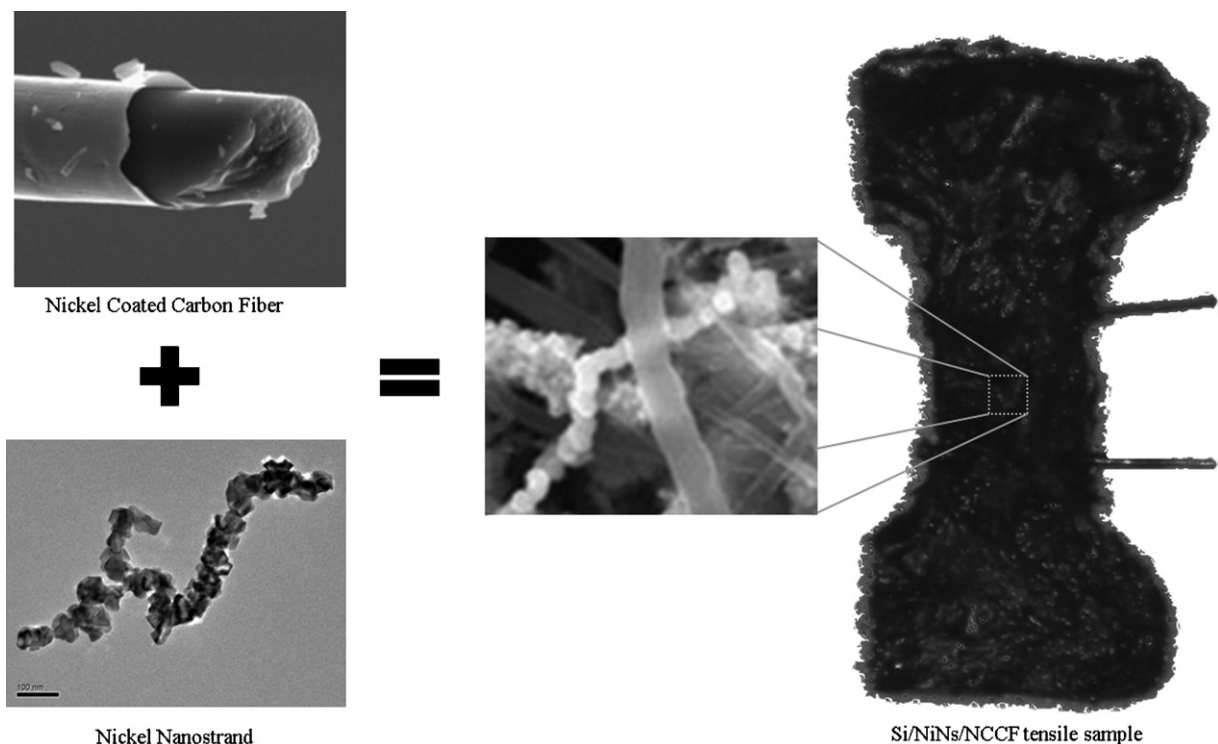


Fig. 6. Material schematic.

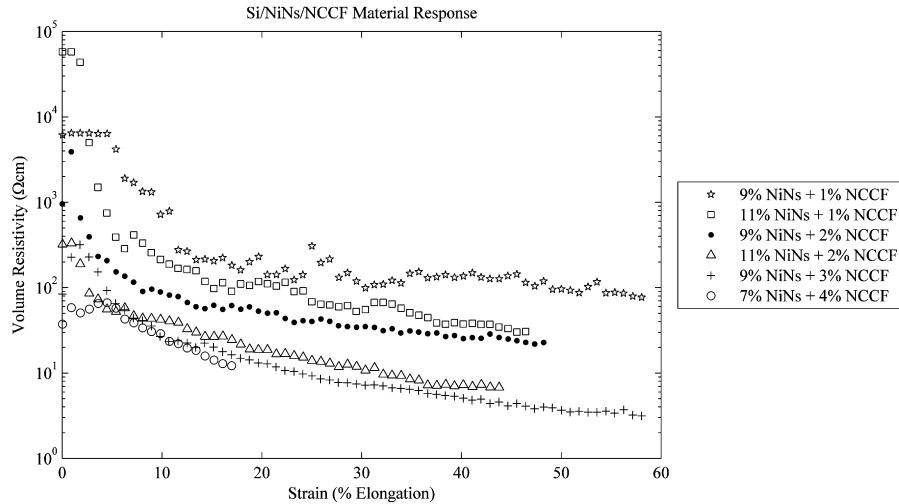


Fig. 7. Piezoresistive response of candidate Si/NiNs/NCCF compositions.

rule and the definition of volume resistivity:

$$\rho = \left(\frac{R_1 V_0}{V_s - V_0} \right) \times \left(\frac{tw}{l} \right) \quad (1)$$

where R_1 is the resistance of the 120Ω series resistor, V_0 the measured voltage signal, V_s the excitation voltage, t the sample thickness, w the sample width, and l the distance between the copper contacts.

4. Results

The response of each of the tested compositions is shown in Fig. 7. In all cases the volume resistivity decreased significantly as the material was strained in tension. Averaged over the three trials for each composition, the 9% NiNs + 1% NCCF and 9% NiNs + 2% NCCF compositions dropped about three-and-a-half orders of magnitude in resistivity; the 11% NiNs + 1% NCCF, about three orders of magnitude; the 9% NiNs + 3% NCCF and 11% NiNs + 2% NCCF, about two-and-a-half orders of magnitude; and the 7% NiNs + 4% NCCF almost one order of magnitude.

Excepting the 7% NiNs + 4% NCCF composition, all of the tested compositions achieved greater than 40% elongation.

5. Discussion

5.1. Performance comparison

The negative gauge factor that was observed shows a diametrically opposite response to that of typical conductive filled polymer composites, but which is characteristic of this nanocomposite system [6,7,15,16]. For clarity, gauge factor is defined by $\Delta R/R = G\epsilon$, where R is electrical resistance and G is the gauge factor. In general, the increase in resistivity with strain of conventional conductive filled polymer composites is thought to be a result of the net increase in distance between conductive filler particles [15,17–19]. However, reports of another elastomer/nickel filament system have been made that show a negative gauge factor, which is attributed to increased filament alignment upon tensile loading [20]. The decrease in resistivity of the Si/NiNs/NCCF system is believed to be largely attributable to the unique bifurcated structure of the

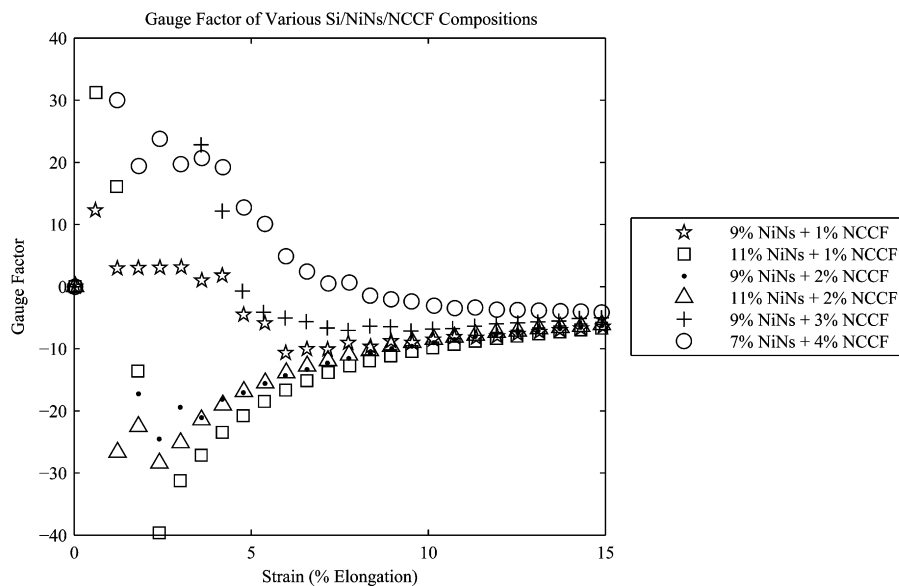


Fig. 8. Comparison of gauge factor, G , as a function of strain for all of the tested compositions. Only 0–15% elongation is shown for clarity and because all compositions approached the same gauge factor value with increasing strain.

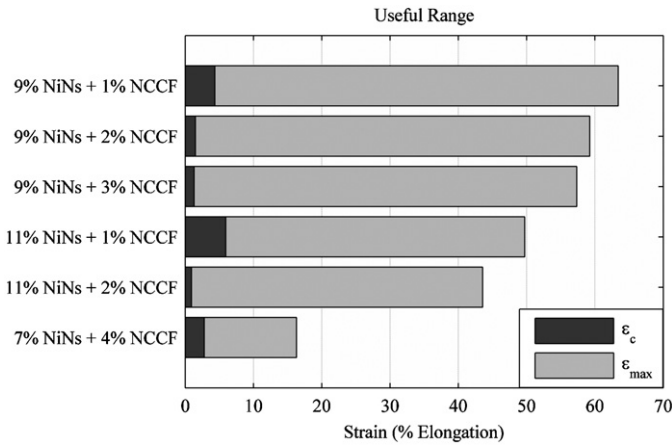


Fig. 9. Comparison of ϵ_c and ϵ_{max} for each composition. The values reported in this figure are the arithmetic means for all trials that were considered.

NiNs particles [7]. In fact, previous work has shown that these nanocomposites have a negative gauge factor in both tension and compression [13].

As is apparent from Fig. 7, when NCCF content is increased there is a dramatic corresponding increase in the overall conductivity of the composite. The composite conductivity is increased about an order of magnitude for each additional volume percent of NCCF.

Increasing NCCF content also tends to decrease the magnitude of the gauge factor, as can be seen from Fig. 8 where the peak gauge factors generally become less and less negative with increasing NCCF content.

The critical strain, ϵ_c , was calculated as the last strain level for which the volume resistivity was within 5% of the maximum volume resistivity. As predicted, ϵ_c did decrease with increasing NCCF content for both the 9% NiNs and 11% NiNs compositions (Fig. 9). Although there was no comparative sample reported to test this trend for the 7% NiNs + 4% NCCF sample, we note that – as stated previously – lower NCCF content samples were tested at 7% NiNs loadings but there was no response. This is to say that ϵ_c was never reached at NCCF content lower than 4% for these samples. This corroborates the results of the other samples because ϵ_c effectively decreased from infinity to a finite value as NCCF content was increased.

Fig. 9 also indicates a general dependence of the maximum elongation on both NiNs and NCCF content. Increasing the volume percentage of either significantly decreases the maximum elongation of the nanocomposite system before fracture. This is thought to be due to the fact that increased filler volume fraction leads to a larger number of inhomogeneities in the composite material. These inhomogeneities act as stress risers and lead to void formation [21]. Thus, increasing either NiNs or NCCF content leads to a statistical increase in void formation probability and an accompanying decrease in the strain required to cause failure. Additionally, the extreme disparity in elastic modulus between the NiNs/NCCFs and the silicone matrix may lead to separation at the filler/matrix interfaces [22], also causing void formation.

5.2. Measurement error

In order to quantify repeatability between gauges, the maximum and minimum resistivity of all trials was calculated for each composition as a function of strain. The smallest possible strain that a measured resistivity could represent was given by the strain at which the mean resistivity was equal to the maximum resistivity. Similarly, the largest possible strain that a measured resistivity could represent was given by the strain at which the mean resistivity

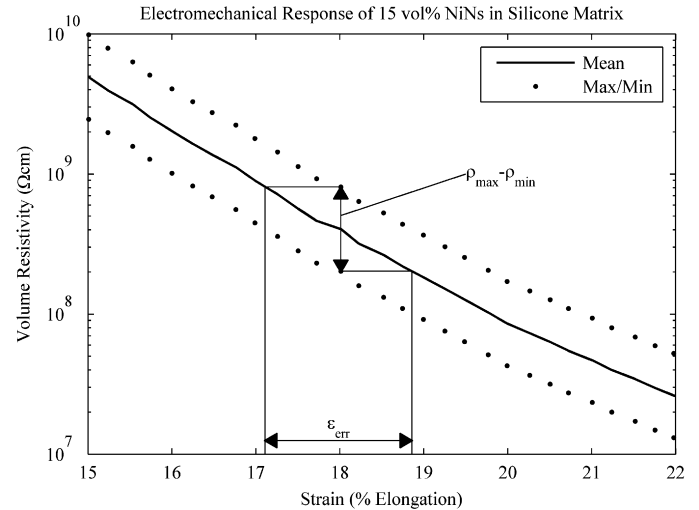


Fig. 10. Schematic representation of how ϵ_{err} was calculated from the range of the volume resistivity for a given strain.

tivity was equal to the minimum resistivity. ϵ_{err} is defined as the difference between these two quantities. This process is illustrated in Fig. 10.

This represents, in essence, the predicted uncertainty in the strain level indicated by the gauge—based on the uncertainty in the measured resistivity signal. The measurement error profile of each candidate composition is shown in Fig. 11.

We note that unlike the previously discussed parameters (ϵ_c , ϵ_{max} , G), ϵ_{err} is not an intrinsic property of the nanocomposite

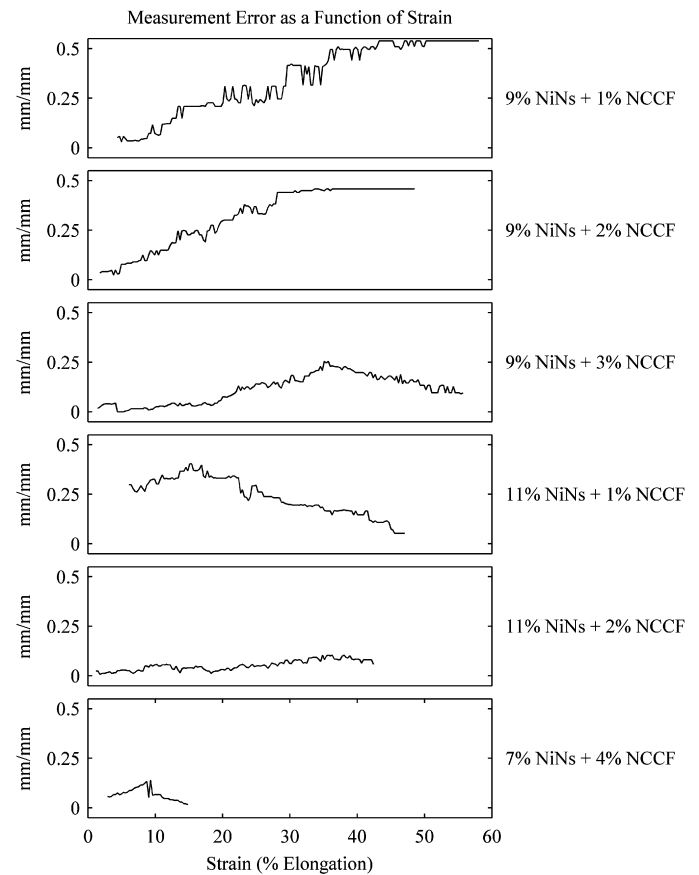


Fig. 11. Measurement error profiles for each candidate composition.

system. It is, rather, a function of process parameters. ε_{err} varies with composition only because certain compositions increase the difficulty of achieving ideal spatial particle distribution. As stated earlier, the viscosity of the pre-cured sample mixture increases with filler content. This can lead to difficulty during the mixing phase of manufacture and consequent non-uniform distribution of particles, which negatively affects the repeatability of the sensors. Additionally, the high viscosity of the more heavily filled samples can be an impediment for the degassing stage of manufacture, leading to porosity in the final samples, which can also adversely affect repeatability. In order to compensate for these problems extra solvent was added. However, excessive solvent may lead to particle settling and a similar problem of non-uniform particle distribution. Thus, uniformity of filler distribution and porosity are the limiting factors in maximizing repeatability. Theoretically there is no barrier to achieving uniform particle distribution and eliminating porosity with any given composition; however, achieving ideal process parameters becomes a practical limitation with certain compositions.

Although processing is the real culprit, some conclusions may be drawn as to the effect of filler content on repeatability (i.e. ease of manufacture). As filler content increases uniformity of particle distribution becomes easier to achieve. This is usually reflected in a decrease in ε_{err} for any given NCCF volume fraction (although at low strains the 11% NiNs + 1% NCCF specimen deviate from this trend). The effect of increasing NCCF content is a similar decrease in ε_{err} , though more pronounced than in the case of varying NiNs volume fraction. This is likely due to the disparity in length scale. As the volume fraction of NCCF reaches a level such that uniform distribution of this phase is practically achievable, the 'super-node' effect created by this phase dominates the influence of non-uniformity in the distribution of the much smaller NiNs phase. As a result the composition with the smallest ε_{err} over the entire tested strain range was found to be the 11% NiNs + 2% NCCF, which had an average of 0.05 mm/mm error.

5.3. Optimization

Based on the experimental tests that were performed an empirical optimization was performed. As stated previously, the optimization problem for this study was to maximize ε_{max} while simultaneously minimizing ε_c , and ε_{err} . As such, an appropriate objective function that satisfies our demands is:

$$f(\text{NiNs vol\%, NCCF vol\%}) = \frac{\varepsilon_{max}}{\varepsilon_c \times \varepsilon_{err}}$$

$$\varepsilon_{max} = \varepsilon_{max}(\text{NiNs vol\%, NCCF vol\%})$$

$$\varepsilon_c = \varepsilon_c(\text{NiNs vol\%, NCCF vol\%})$$

$$\varepsilon_{err} = \varepsilon_{err}(\text{NiNs vol\%, NCCF vol\%})$$
(2)

And the optimization problem is formulated as:

$$\max_{x \in \{7,9,11\}, y \in \{1,2,3,4\}} (f)$$

$$\text{subject to}$$

$$\varepsilon_{max} \geq 0.4$$
(3)

where x = NiNs vol %, and y = NCCF vol %. The optimization algorithm consisted in the following: (1) measure ε_{max} , ε_c , ε_{err} ; (2) compute the value of the objective function, f ; and (3) choose the composition which maximizes the objective function.

Table 1 shows the values of each of the parameters as well as the value of the objective function. From Table 1 it can be seen that the best performing composition was the 11% NiNs + 2% NCCF. The value of f for the 7% NiNs + 4% NCCF composition put it in 4th place numerically; however, the maximum elongation was less than the imposed constraint of 40% and it is therefore thrown out.

Table 1
Summary of composition performance.

Composition	ε_{max} (mm/mm)	ε_c (mm/mm)	ε_{err} (mm/mm)	f
9% NiNs + 1% NCCF	0.63	0.04	0.35	41.88
11% NiNs + 1% NCCF	0.50	0.06	0.24	34.99
9% NiNs + 2% NCCF	0.59	0.02	0.32	121.91
11% NiNs + 2% NCCF	0.44	0.01	0.05	910.45
9% NiNs + 3% NCCF	0.57	0.01	0.11	391.27
7% NiNs + 4% NCCF	0.16	0.03	0.07	N/A

The 9% NiNs + 3% NCCF also performed well, however the repeatability of measurements as given by ε_{err} was about half that of the 11% NiNs + 2% NCCF composition. With improved control of process parameters it is believed that the repeatability for this composition can be enhanced. It is therefore suggested that both of these compositions be further developed for application as a large strain sensor material.

The optimized nanocomposite composition of 11% NiNs + 2% NCCF is capable of measuring strains from as little as 1% up to as large as 44% elongation with an average error of only $\pm 2.5\%$ elongation.

6. Conclusion

In this study it has been shown that the piezoresistive material response of Si/NiNs/NCCF can be tuned by varying the volume percentage of NiNs and NCCF. Increasing the volume percentage of NiNs in the composite was found to cause a decrease in ε_c , and improved repeatability between gauges. It was also found that increasing NCCF content caused a decrease in gauge factor magnitude and ε_{max} , and an increase in repeatability and bulk composite conductivity.

From these results, the composition that showed the most promise for use as a large displacement sensor was determined to be the 11% NiNs + 2% NCCF because of its large maximum elongation (ε_{max}), low critical strain (ε_c), and minimal measurement error (ε_{err}). This material is capable of measuring strains from 1% to 44% elongation. This is over 8 times greater than the maximum strain measurable using traditional foil strain gauges [16]. The average measurement error of this composition was 0.05 mm/mm (± 0.025 mm/mm) and may be improved by better control of process parameters.

The results of this study indicate that Si/NiNs/NCCF nanocomposites in concentrations of 11 vol% NiNs + 2 vol% NCCF can be used as simple, inexpensive large displacement sensors that can measure strains over 40% elongation.

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George Hansen is the Founder and President of Conductive Composites Company, where nanostrands are developed and manufactured. With a B.S. in chemical engineering, his central current interest is in the electromagnetic modification of polymers and composites with nanomaterials. He has been the Principal Investigator for nine major government awards in the field, totaling over \$9 million in funding. He is the recipient of an R&D 100 Award, a NASA Nanotech 50 Award and is the 2010 Utah Innovator of the Year. Most recently, he is advancing these materials into full manufacturing.